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09/408,323	09/29/1999	JERRY R. EBNER	MTC6610(39-2	3903

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ST LOUIS, MO 63102

EXAMINER

MAIER, LEIGH C

ART UNIT	PAPER NUMBER
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1623

DATE MAILED: 06/18/2002

17

Please find below and/or attached an Office communication concerning this application or proceeding.

# Office Action Summary

Application No.

09/408,323

Applicant(s)

Ebner et al

Examiner

Leigh Maier

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— The MAILING DATE of this communication appears on the cover sheet with the correspondence address —

## Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136 (a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

## Status

- 1) ☒ Responsive to communication(s) filed on Mar 25, 2002
- 2a) ☐ This action is FINAL. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 35 C.D. 11; 453 O.G. 213.

## Disposition of Claims

- 4) ☒ Claim(s) 1-244 and 247-300 is/are pending in the application.
- 4a) Of the above, claim(s) \_\_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-244 and 247-300 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.
- 8) ☐ Claims \_\_\_\_\_ are subject to restriction and/or election requirements.

## Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_\_ is/are a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
- 11) ☐ The proposed drawing correction filed on \_\_\_\_\_ is: a) ☐ approved b) ☐ disapproved by the Examiner.  
If approved, corrected drawings are required in reply to this Office action.
- 12) ☐ The oath or declaration is objected to by the Examiner.

## Priority under 35 U.S.C. §§ 119 and 120

- 13) ☐ Acknowledgement is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).  
a) ☐ All b) ☐ Some\* c) ☐ None of:  
1. ☐ Certified copies of the priority documents have been received.  
2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.  
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).  
\*See the attached detailed Office action for a list of the certified copies not received.
- 14) ☒ Acknowledgement is made of a claim for domestic priority under 35 U.S.C. § 119(e).  
a) ☐ The translation of the foreign language provisional application has been received.
- 15) ☐ Acknowledgement is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121.

## Attachment(s)

- 1) ☐ Notice of References Cited (PTO-892) 4) ☒ Interview Summary (PTO-413) Paper No(s). \_\_\_\_\_
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948) 5) ☐ Notice of Informal Patent Application (PTO-152)
- 3) ☒ Information Disclosure Statement(s) (PTO-1449) Paper No(s). 12, 16 6) ☐ Other: \_\_\_\_\_

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## **DETAILED ACTION**

### ***Status of the Claims***

Claims 23, 54, 150-153, 242-244, and 248 have been amended. Claims 248-300 have been added. Claims 245 and 246 have been canceled. Claims 1-244 and 247-300 are pending. Any objection or rejection not expressly repeated has been withdrawn. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Applicant's arguments with respect to claims 1-244 and 247 have been considered and will be addressed as they apply to the current grounds of rejection.

### ***Continued Examination Under 37 CFR 1.114***

A request for continued examination under 37 CFR 1.114, including the fee set forth in 37 CFR 1.17(e), was filed in this application after final rejection. Since this application is eligible for continued examination under 37 CFR 1.114, and the fee set forth in 37 CFR 1.17(e) has been timely paid, the finality of the previous Office action has been withdrawn pursuant to 37 CFR 1.114. Applicant's submission filed on March 25, 2002 has been entered.

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***Information Disclosure Statement***

In Applicant's response, filed March 25, 2002, it was noted that an initialed copy of PTO/SB/08A, corresponding to the fourth IDS did not accompany the last Office action, paper no. 11. At the time the last action was mailed out, the fourth IDS had not yet been placed in the file. Applicant will find initialed copies of the fourth and fifth IDS appended hereto.

***Claim Objections***

Claims 252, 259, and 266 are objected to because of the following informalities: These claims have a small rectangle where it appears a degree symbol (°) should be. Appropriate correction is required.

***Claim Rejections - 35 U.S.C. § 112***

Claims 10, 11, 117-199 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Claim 10 recites the limitation "the reagent" in line 5. There is insufficient antecedent basis for this limitation in the claim. The claim is thus rendered vague and indefinite.

Claim 11 recites the limitation "the solution or slurry" in lines 2-3. There is insufficient antecedent basis for this limitation in the claim. The claim is thus rendered vague and indefinite.

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Claim 117 recites a limitation regarding the process set forth in claim 100 that is incomprehensible. The claim recites “. . . continuously contacting *another* aqueous reaction mixture containing said product, formaldehyde and formic acid *produced in said first aqueous reaction medium . . .*” (Emphasis added) It appears that “the first reaction mixture” and “the another reaction mixture” are the same reaction mixture. The claim goes on to recite a reduced rate of oxygen feed. It is not clear if the claim is requires (1) merely reducing oxygen flow later in the process; (2) having two separate reactors conducting the same process, but with reduced oxygen flow in one; or (3) something else. The claim is rendered vague and indefinite. The dependent claims do not clarify this claim.

***Claim Rejections - 35 U.S.C. § 103***

Claims 1-34, 49-52, 200-229, 248-251, 256-258, 263-265, 270, 271, 288, and 289 are rejected under 35 U.S.C. 103(a) as being unpatentable over NITROKEMIA (EP 019445) in view of HERSHMAN et al (US 4,264,776), VAN DAM et al (J. Catalysis, 1991), and CHOU (US 4,624,937).

*Regarding claims 1-12, 200-223, 248-251:* The claims are drawn to a process for the oxidative removal of a carboxymethyl group from the tertiary amine, N-phosphonomethyliminodiacetic acid (PMIDA), to prepare N-(phosphonomethyl)glycine (glyphosate) with an oxidation catalyst (noble metal on carbon) in the presence of oxygen. (Claims 248-251 are drawn to a generic oxidation process.) The claims recite functional

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limitations defining it as what the disclosure terms “deeply reduced.” Claims 1, 3-5, 248, and 250 recite a functional limitation regarding CO desorption properties of the catalyst. Other dependent claims recite limitations regarding the use of a continuous reactor system, the rate of oxygen use, introduction of sacrificial reducing agents, and recycling the catalyst.

*Regarding claims 13-32, 206-217, 256-258, and 263-265:* These claims are drawn the same process as set forth above, but reciting different functional limitations of the reduced catalysts. These limitations are the ratio of carbon to oxygen atoms at the surface of the catalyst and the ratio of oxygen to noble metal atoms at the surface of the catalyst.

*Regarding claims 33, 34, 218-223, 270, and 271:* These claims require heating the catalyst to at least 400°C after deposition of the noble metal on the carbon surface.

*Regarding claims 49-52, 224-229, 288, and 289:* These claims require exposure of the catalyst to a reducing atmosphere (particularly sodium borohydride or ammonia) after deposition of the noble metal on the carbon surface.

NITROKEMIA discloses the oxidation of N-phosphonomethyliminodiacetic acid (PMIDA) with an oxidation catalyst (noble metal on carbon) in the presence of oxygen in an acidic solution to prepare glyphosate, a secondary amine. See examples. Formic acid and formaldehyde are introduced into the reaction mixture, as they are known reaction side products of this reaction.

NITROKEMIA discloses the repetitive use of the catalyst for the preparation of glyphosate from PMIDA. The process comprises conducting the process and separating the product from the

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catalyst by filtration and re-using the catalyst for subsequent cycles. The reference further teaches that the catalytic activity of the recovered catalyst does not significantly decrease even after 10 working cycles. See col 4, lines 33-55.

NITROKEMIA uses a commercial catalyst and does not specifically teach any recited steps to produce a “reduced” catalyst and likewise does not characterize the catalyst with regard to the CO desorption. Neither does the reference teach a continuous process. The reference teaches preferred oxygen pressure, (see page 7, first full paragraph) but not specifically in terms of rate of “oxygen utilization.”

HERSHMAN teaches the production of secondary amines from catalytic oxidation of tertiary amines in the presence of oxygen. See abstract. The reference teaches that the activated carbon alone will catalyze the reaction, but the use of a noble metal produces a more rapid reaction. See col 7, lines 54-68. The reference exemplifies the use of Rh/C and Pt/C as the catalyst. See Table 2. The catalyst is prepared by activation at high temperature (800°-900°C) of a carbon support followed by impregnation of support with the noble metal and reduction (with sodium borohydride, for example) of the thus formed noble metal/carbon catalyst. See col 4, lines 44-57 and col 8, lines 8-19. The reference also teaches a continuous oxidation process and discusses oxygen pressures and flow rates. See col 2, lines 46-60; col 3, lines 6-10; and the examples.

VAN DAM teaches the preparation of catalysts comprising platinum on carbon. The reference teaches the reduction step (after dispersal of Pt on carbon surface) at about 400°C and

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the use of other reducing agents, such as formaldehyde and hydrogen gas. See page 336, right hand column.

CHOU teaches an improved (relative to HERSHMAN) two-step protocol for the activation of a carbon support surface comprising (1) treatment of the surface with an oxidizing agent, such as nitric acid, nitrogen dioxide,  $\text{CrO}_3$ , hypochlorite, and oxygen gas, and (2) pyrolysis at temperatures, preferably from about  $800^\circ$  to  $1200^\circ\text{C}$ . Regarding the pyrolysis, the reference specifically teaches the use of a pyrolyzer apparatus at  $930^\circ$  and that the use of an atmosphere containing  $\text{NH}_3$  produces a more active catalyst. See col 4-5.

It would have been obvious to one of ordinary skill at the time the invention was made to prepare glyphosate in a continuous process with a reduced catalyst comprising a noble metal on carbon, wherein the reduced catalyst would be produced by the process taught by the combination of HERSHMAN, VAN DAM, and CHOU. This combination of references teaches the reducing process used to produce the catalysts described in, for example, Table 1 and Table 5 of the instant disclosure. The treatment of the same materials by the same process must produce the same physical characteristics. In this case, (1) the CO desorption properties; (2) C/O ratios; and (3) O/noble metal ratios. The artisan would be motivated to use a reduced catalyst as taught by the combination of HERSHMAN, VAN DAM, and CHOU for its increased activity. One of ordinary skill would be motivated to conduct a continuous process, as HERSHMAN had taught its utility. Regarding claim 51, neither HERSHMAN nor van DAM teach ammonia for reducing the catalyst after deposition of noble metal. However, it would be within the scope of the artisan



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to select another reducing agent as a functional equivalent to those taught by those references.

The artisan would reasonably expect success in using ammonia, as CHOU had taught its utility in reducing the carbon surface in the two-step process taught in that reference.

Regarding oxygen utilization, pressures and flow rates are discussed both in NITROKEMIA and HERSHMAN. It would be within the scope of the artisan to optimize variable through routine experimentation.

It would be further obvious to recycle the catalyst, as NITROKEMIA had taught the feasibility thereof. The artisan would be motivated to do this for economic reasons.

Finally, even if “sacrificial reducing agents” were not automatically introduced by virtue of being side products, it would be obvious to do so in order to keep the surface reduced, as taught by HERSHMAN and VAN DAM.

Claims 100-143 and 242-247 are rejected under 35 U.S.C. 103(a) as being unpatentable over NITROKEMIA (EP 019445) in view of HERSHMAN et al (US 4,264,776), VAN DAM et al (J. Catalysis, 1991), and CHOU (US 4,624,937) and further in view of JOHNSON et al (US 6,153,753).

The claims are drawn to the same general oxidation process as described above. This set of claims require a continuous process in a stirred tank reactor. Dependents are drawn to catalyst characteristics, including limiting the catalyst to Pt/C, oxygen feed rate, reactor residence time, recycling the catalyst, isolation of the product, and the use of sacrificial reducing agent.

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Dependent claims also appear to require at least two reaction stages, but exactly what is required is unclear, as discussed above.

NITROKEMIA, HERSHMAN, VAN DAM, and CHOU teach as set forth above. The preparation of reduced catalysts comprising noble metal, particularly platinum, on carbon and their use in the continuous oxidation of PMIDA to prepare glyphosate. Noble metal on carbon catalysts are known to be able to reduce formaldehyde. The references do not teach the use of a stirred tank reactor.

JOHNSON teaches the use of Pt/C catalysts for the oxidative preparation of secondary amines from tertiary amines, including glyphosate from PMIDA, in continuous stirred tank reactors. See col 19, lines 7-15 and col 50-52. See also the schemes in the reference. Noble metal on carbon catalysts are known to reduce formaldehyde.

It would have been obvious to one having ordinary skill in the art at the time the invention was made to prepare the reduced catalysts as discussed above to prepare glyphosate from PMIDA in a continuous stirred tank reactor because JOHNSON had taught the utility of this type of reactor for oxidation reactions comprising noble metal on carbon catalysts. In using these catalysts, made obvious from the above references, for this process, one of ordinary skill would necessarily accomplish the conditions such as formaldehyde wt% content and noble metal dissolution set forth in the claims. It would be within the scope of the artisan to optimize result effective variables, such as reagent conversion/reaction stage, oxygen feed rate, temperature, and reactor residence time through routine experimentation. Multi-stage reactors are common in

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continuous processes. It would be within the scope of the artisan to optimize the number of reaction stages based on yields and costs. The use of sacrificial reducing agents, recycling the catalyst and product isolation by filtering the catalyst have been addressed above.

Claims 1-99, 200-241, and 248-300 are rejected under 35 U.S.C. 103(a) as being unpatentable over in view of NITROKEMIA (EP 019445) in view of JALAN et al (US 4,186,110), ITOH (US 5,876,867), HERSHMAN et al (US 4,264,776), and CHOU (US 4,624,937).

The claims are drawn to a process as set forth above. See above for discussion of claims not outlined below.

*Regarding claims 33-48:* These claims require heating the catalyst at temperatures of at least 400°C, and up to about 1200°C, after deposition of the noble metal on the carbon surface.

*Regarding claims 53-99, 230-241, 259-262, 266-269, and 270-300:* These claims require the use of a catalyst, as set forth above, further comprising a promoter which may or may not be alloyed with the noble metal at the surface. Dependent claims promoter species, weight percentages thereof, and catalyst functional limitations (CO desorption, C/O ratios, O/M ratios) as set forth above.

NITROKEMIA teaches as set forth above.

HERSHMAN teaches as set forth above.

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NITROKEMIA does not teach the use of a noble metal/carbon catalyst comprising one or more promoters in which the promoter may or may not be alloyed with the noble metal.

JALAN teaches noble metal alloys (alloys with other noble metals or with a variety of base metals, such as Ti and Al), supported on carbon, as catalysts. JALAN teaches that particular steps in the preparation of these catalysts, including heating to temperatures ranging up to about 1000°C in a reducing atmosphere. See particularly the abstract, the examples, and col 5, line 51-68. Although the discussion in JALAN centers on the use of the catalysts in fuel cells, JALAN expressly teaches that these catalysts also find application in chemical processes. See sentence bridging col 3 and 4.

ITOH teaches similar catalysts to JALAN which are prepared at a high temperature in a reducing atmosphere to alloy a wide variety of base metals, such as Ga, V, Cr, Mn, Fe, Co, Ni, Cu, with platinum on a carbon support. ITOH teaches that catalysts thus prepared have a longer lifetime due to less leaching out of noble metal from the catalyst. See abstract and col 2-3.

It would have been obvious to one having ordinary skill in the art to have altered the catalysts of NITROKEMIA by the process of JALAN, by heating the catalyst in a reducing environment to prepare a catalyst comprising an alloyed promoter, for the continuous preparation of glyphosate from PMIDA for the increased activity and increased acid resistance imparted onto the catalyst by this process. It would further be within the scope of the artisan to determine the optimum promoter metal and concentration thereof through routine experimentation.

Furthermore, in view of HERSHMAN and CHOU, it would have been obvious to alter the

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process of JALAN or ITOH by first activating the carbon support in a reducing atmosphere before deposition of the noble metal, as set forth above.

In paper no. 14, Applicant has submitted arguments pertaining to the motivation for using catalysts taught by JALAN and/or ITOH in the instant process because JALAN and ITOH are primarily concerned with fuel cells, and there is no teaching in any of these references of a chemical oxidation reaction in a system which can "solubilize a noble metal." This argument is not persuasive. The examiner agrees that these references are primarily concerned with fuel cells: *acid* fuel cells. Given that these alloyed catalysts are taught to have increased catalytic activity and acid resistance *and* are useful for chemical catalysis, and HERSHMAN has specifically noted the problem of noble metal leaching in acidic solution (see col 8, lines 4-8 and example 3), the artisan would be motivated to use them for the instant oxidation for the disclosed advantages.

Claims 100-199, 242-244, and 247 are rejected under 35 U.S.C. 103(a) as being unpatentable over in view of NITROKEMIA (EP 019445) in view of JALAN et al (US 4,186,110), ITOH (US 5,876,867), HERSHMAN et al (US 4,264,776), and CHOU (US 4,624,937) and further in view of JOHNSON et al (US 6,153,753).

The process of claims 100-143, 242-244, and 247 are as set forth above. Dependent claims require the use of a catalyst comprising a promoter which may or may not be alloyed to the noble metal. The catalysts have functional limitations regarding CO desorption, C/O ratio,

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and O/M ratios as discussed above. Other functional limitations pertain to catalyst particle size and pore volume.

The references teach as set forth above.

The use of noble metal on carbon catalysts for the oxidation of PMIDA to prepare glyphosate in a continuous stirred tank reactor is discussed above. It would have been obvious to one of ordinary skill in the art at the time the invention was made to use alloyed catalysts, as taught by JALAN or ITOH, with surface activation, as taught by HERSHMAN and CHOU for the advantages discussed above. In preparing the catalysts as taught, and using them in the oxidation of PMIDA to prepare glyphosate, the ordinarily skilled worker would necessarily produce catalysts having the functional limitations recited in the claims. As noted above, it would be within the scope of the artisan to optimize the promoter and wt% thereof through routine experimentation.

### ***Double Patenting***

Claims 57, 91, 144, 274, and 292 are provisionally rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over claim 54 of copending Application No. 09/408,296. Although the conflicting claims are not identical, they are not patentably distinct from each other because the instant processes are drawn to the oxidation of (1) PMIDA or salt thereof to glyphosate or a salt thereof using a catalyst comprising a noble metal on carbon and a promoter; or (2) any reagent using a catalyst comprising a noble metal on carbon

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and a promoter. The instant processes are not patentably distinct because 09/408,296 is drawn to the use of the same catalyst for the oxidation of the single reagent, salt of PMIDA, to the single product, salt of glyphosate, expressly encompassed by the instant claims.

This is a provisional obviousness-type double patenting rejection because the conflicting claims have not in fact been patented.

### ***Conclusion***

The instant process claims fall into two general categories--those requiring a catalyst comprising a promoter and those not requiring a promoter. It is noted that claims for a catalyst have been allowed in the parent application, serial number 09/248,655. A review of the allowed claims find that they all require a promoter. While many of the instant process claims require a catalyst comprising a promoter which is similar to the catalyst in the allowed claims, none appear to be commensurate in scope at this time. Process claims amended to require a catalyst having all the limitations of the allowed catalyst will be allowable.

### ***Examiner's hours, phone & fax numbers***

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Leigh Maier whose telephone number is (703) 308-4525. The examiner can normally be reached on Tuesday, Wednesday, or Friday 7:00 to 3:30 (ET).

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Mr. Johann Richter (703) 308-4532, may be contacted. The fax phone number for Group 1600, Art Unit 1623 is (703) 308-4556 or 305-3592.

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Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the Technology Center 1600 receptionist whose telephone number is (703) 308-1235.

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A handwritten signature in black ink, appearing to read "Leigh C. Maier", with a long horizontal flourish extending to the right.

Leigh C. Maier  
Patent Examiner  
June 14, 2002